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# Process for Particulate Material

The present invention relates to a process for preparing a particulate material based on recycled paper waste sludge containing cellulose fibres, china clay and calcium carbonate, and products prepared therefrom.

There is an increasing industry in recycling waste paper. Waste paper is increasingly collected throughout the UK with intent to recycle it. However, the term "waste paper" includes a myriad of different types of paper-fibre materials, all of which are provided by the public in the belief that they are all 'recyclable' in the same way.

A number of the "waste papers" also contain materials that have been added during the original paper production and conversion processes, and these must be removed to provide a clean fibre material

suitable for re-use in a paper machine. Two main materials that need to be removed from many types of waste papers are china clay, also known as kaolin, and calcium carbonate. These are added to certain paper products to make the paper opaque and to improve its printing quality. Other materials that commonly need to be removed from waste papers are staples, clips, glues, plastic coatings, etc.

Large separated materials from waste papers are generally disposed of directly as "trash". These include plastics, plastic coating materials and metal clips, etc. Fine separated materials are carried in an aqueous suspension to an effluent treatment process where they are removed by gravity sedimentation, and then further dewatered to 40-60% solids for disposal. This creates a biological sludge, (which can also be mixed with a primary sludge before the dewatering stage).

The "paper waste sludge solids" are currently simply deposited in landfill sites or spread on land. However, increasing environmental legislation requires this depositing to be reduced.

Meanwhile, paper making processes generally also create a paper-fibre based sludge waste product which is also currently deposited in landfill sites or spread on land.

A typical quantity of waste sludge from a paper recycling and paper making facility can be 250

tonnes per day, representing a significant amount of waste sludge material.

It is an object of the present invention to provide a process for treating such waste material to form an industrial useful product.

Thus, according to one aspect of the present invention, there is provided a process for preparing a particulate solid material comprising the steps of:

(a) obtaining a paper-fibre waste solid material having a ratio of china clay, or equivalent, to chalk, or equivalent, greater than a pre-determined minimum;

(b) treating the material to reduce the moisture content and form a granular material; and

(c) calcining the granular material at a temperature of approximately 1300°C or higher to provide a particulate, 100% solids, material.

The paper-fibre waste solid material can be provided by the non-hazardous waste material arising from the recycling of waste paper and tissue. Such material is generally in the form of sludge, having a moisture content of over 45%, and commonly over 55%, 60%, or higher. Such sludge contains china clay, calcium carbonate as well as the general cellulose fibre content. Such material may also

include surplus biomass from biological effluent treatment processes and water treatment processes which produce sludge. Minor components may include non-fibrous 'contraries' materials arising from waste paper, including such items as polythene, plastics, metal (in the form of wire, staples, paper clips).

The term china clay or equivalent includes any form of hydrated aluminium silicate, including kandites, kaolins and the like.

The term chalk or equivalent includes any form of calcium carbonate, which includes the forms of limestone.

The sludge is preferably dewatered so as to produce a sludge having an increased solids content.

Analysis of the china clay:chalk ratio is preferably carried out prior to the dewatering of the waste material.

One method of analysis is termed 'acid extraction'. A sample of sludge cake of known dry solids is treated with 10% acetic acid solution to dissolve calcium carbonate. The remaining solids are filtered out, washed, dried at 105°C and weighed. The loss in weight determines the calcium carbonate content of the dry solids content of the sludge cake. The remaining solids are further heated to a high temperature such as, but not limited to, 800°C,

to form an ash free from carbon which again is weighed. The further loss in weight determines the cellulose content of the sludge cake. The remaining weight of ash determines the clay content. From these calculated values the clay:chalk (calcium carbonate) ratio is calculated.

By the acid extraction method, the pre-determined minimum is approximately 0.2.

Another method of analysis is termed "ash/acid extraction". For this, a weighed sample of dried sludge cake is treated to 800°C to form an ash free from carbon. The cooled sample is weighed and the loss in weight determines the cellulose content plus the carbon dioxide arising from the destruction of the calcium carbonate. The cooled sample is then treated with 10% acetic acid, filtered, washed, dried and weighed. The further loss in weight determines the calcium oxide content and the remaining weight determines the clay content. From these measured values the cellulose, calcium carbonate and clay contents can be calculated.

By the ash/acid extraction method, the pre-determined minimum is approximately 0.13.

Dewatering is a process well known in the art, as are the process parameters for the pressing action. Traditionally, a polyelectrolyte is added to a waste material, which material can often have only a 7% solids content, in order to agglomerate the very

fine waste material, commonly termed "fines", in the process. The dewatering process increases the solids content several fold, such as to a typical solids content of 45%.

Where the china clay:chalk ratio is found to be less than the pre-determined minimum in the waste material, an embodiment of the present invention is to add a conditioning material to the waste material. The conditioning material is preferably partly, substantially or wholly china clay, or at least a china clay suspension, or another silicate material having the same properties. A dispersing agent could also added to the conditioning material in order to maintain the china clay or similar material in a suspended form, in a liquid host such as water.

It has been found that by varying the conditioning material content in the waste material, the dewatering process can result in a material having less solids content than, for example 45%, such content being even 22% or lower.

In an embodiment of the present invention, the conditioning material is added to the waste material even when the china clay:chalk ratio is greater than the pre-determined minimum, in order to effect the properties of the material as treated thereafter.

The dewatering process provides a sludge material having a solids content generally in the range 22-55%.

In another embodiment of the present invention, the ratio of silica and aluminium to natural fillers in the paper sludge is based on the addition of the conditioning material which uses silica and/or aluminium to promote fluxing of the paper sludge.

The treatment step (b) of the present invention could be carried out by operating on the paper-fibre waste material in various ways, such as compression, extrusion or the like, and/or a combination of such processes. Extrusion through a die with apertures produces lines of a material which either inherently or by further processing produces a granulated material. A granulating press could be used for this step (b).

The treatment step (b) of the present invention could also be provided by direct heat contact, such as conduction. A heat transfer material could be used such as steam, etc. in this regard. The action of the heat treatment is to partly or substantially 'dry' the paper-fibre based material.

Such a heat treatment process could be carried out with agitation, such as provided by a rotary apparatus such as a rotary dryer. An inclined rotary processor can reduce water content while inducing a tumbling action against heated surfaces

at controlled temperatures. Preferably, any heat treating apparatus allows for a wholly or substantially continuous feed of starting material. The process may involve recycling of material to adjust the range of particle sizes of granular material produced.

The treatment step is preferably carried out at a raised temperature, preferably between 60-80°C, although not limited thereto.

The treatment step could also be carried out under an inert atmosphere. Such an inert atmosphere could be provided by displacing air with steam, either by direct injection or by evaporated water. Heat treating the material in an inert atmosphere reduces the moisture content and forms the material into rounded granules approximately 3mm-30mm in size.

In another embodiment of the present invention, the granular material formed by the treatment step could be further granulated, that is further processed in a granulator or the like to better form a more regular, generally spherical, shaped solids material.

The material formed by the treatment step preferably has a solids content in the range of approximately 45-90% solids. It has been found that the moisture content of the so-formed granular material affects the size of the particulate material formed in the subsequent calcining step.

The calcining of the granular material is adapted to reduce the moisture in the material to zero. The particulate material being formed also becomes porous, either partly or substantially, by the burning of the cellulose content of the waste material.

The calcining also fuses the granular material, so as to provide solid pellets.

Preferably, the granular material is calcined with agitation, such as provided by a rotary apparatus, an example is a high temperature rotary furnace, such as a tube. The rotary action serves to provide a more even evaporation of moisture and burning of cellulose.

Preferably, the calcining temperature is greater than 1300°C, possibly approximately 1320°C or higher.

In one embodiment of the present invention, the calcined product is reduced, for example granulated or refined or otherwise ground. With certain size reduction, such as milling or the like, a particular particle maximum size or range of sizes can be achieved. For example, through milling the calcined particulate material to have a maximum particle size of 100μm, a fine particulate material is formed which is useable as a cementious material, for example as a partial, substantial or complete replacement or substitute for current cement

materials in building blocks such as 'breeze blocks' and the like.

According to a second aspect of the present invention, there is provided a particulate solid material whenever prepared by a process as herein described.

The particulate solid material preferably has a bulk density of less than 1,000kg/$m^3$, and generally in the range 560 kg/$m^3$ to 800 kg/$m^3$. The particulate solid material is preferably in the form of an aggregate. Preferably, the aggregate has a mean particle size of between 3-15mm. The mean particle size can be favoured based on the moisture content of the material made by the treatment step. The use of a drier material provides a smaller mean particle size, whereas the use of wetter material provides a larger mean particle size.

The particulate solid material may be usable in a number of industrial applications, including as a light weight aggregate for making cementitious, concrete or other building blocks, or as a replacement or filler material in other building applications. The material is also 'eco-friendly'. Examples of the present invention will now be described by way of example only.

Example 1

The starting material was provided from waste from paper recycling in the form of raw separated solids with water or sludge from biological or chemical treatment of the separated solids. The starting material included china clay, calcium carbonate, cellulose fibres and a water content of approximately 93%.

The material was analysed by the acid extraction procedure to determine the ratio of clay:chalk and to further determine the amount of conditioning material to be added to bring the clay:chalk ratio up to 0.2.

The conditioning material may include clay or chalk and is prepared as a suspension in water at a suitable concentration to facilitate pumping.

The material was fed to dewatering equipment with conditioning material injected into the feed pipeline. Dewatered material was collected from the dewatering equipment with water content varying from 55% to 80%.

The dewatered material was fed at 10-15 kg/h into a drier where heat transfer by contact with hot surfaces evaporated water, formed the material into rounded granules and provided an inert atmosphere. The rounded granules were from 3-30mm in size, with occasional granules up to 50mm in size, with a water content of approximately 50%.

The dried material was fed at approximately 15 kg/h into a calciner comprising a rotating tube lined with refractory material and equipped with a propane burner. The calciner was inclined so that material passed counter current to the gas flow, passed through a hot zone at 1300°C-1350°C before being discharged into a receiving tray. The residence time in the calciner was approximately 15-20 minutes and the final processed material was hard to the touch, light brown with a speckled surface in appearance and produced a clear sound when dropped onto a hard surface.

Example 2

The starting material was provided from waste from paper recycling in the form of raw separated solids with water or sludge from biological or chemical treatment of the separated solids. The starting material included china clay, calcium carbonate, cellulose fibres and a water content of approximately 93%.

The material was analysed by the acid extraction procedure to determine the ratio of clay:chalk and to further determine the amount of conditioning material required to bring the clay:chalk ratio to >0.2.

The conditioning material may include clay or chalk and in this example was presented as a filter cake

which was prepared as a suspension in water at a suitable concentration to facilitate pumping.

The material was fed to dewatering equipment with the conditioning material injected into the feed pipeline. Dewatered material was collected with water content varying from between 40% and 45%.

The dewatered material was fed to a granulating press fitted with an extrusion plate thus producing extruded particles of approximately 20mm diameter having a cylindrical form.

The extruded particles were transferred to a granulating table comprising an inclined surface with a rim to retain material, and inclined at an angle so that rotation of the table produced rounded particles. Small quantities of water were sprayed onto the particles to assist with rounding of them. Undersized and fine particles were separated by screening and were returned to the granulating press to be reformed.

The rounded particles were fed at a rate up to approximately 35 kg/h into a calciner comprising a rotating tube lined with refractory material and equipped with a gas burner. The calciner was inclined so that material passed counter current to the gas flow and passed through a hot zone before being discharged into a receiving tray. The final processed material was hard to the touch and light brown with a speckled surface in appearance. The

bulk density of the product varied from 560 kg/m$^3$ to 920 kg/m$^3$ by adjusting the feed rate and speed of rotation of the calciner.

Example 3

1,130 kg of the final processed material was further treated to reduce its particle size by milling whereby it was passed it through a rotating cylinder containing heavy balls of steel such that the tumbling action of the balls crushed the final processed material to a particle size of less than 100 μm. The milled material was tested by incorporating it in the formulation of concrete products, such as concrete blocks for building. In tests a substantial part of the cement content of the formulation was replaced by the milled material which produced satisfactory concrete blocks having sufficiently similar properties to existing 'breeze blocks' so as to be direct substitute therefor.

The present invention provides an eco-friendly method of using a significant waste product, that is currently simply deposited in landfill sites or spread on land. The process produces a material which is usable in a number of industrial applications, thereby not only increasing the recyclability of waste papers, but provided a beneficial product.